IS 101-4-1 (1988): Methods of Sampling and Test for Paints, Varnishes and Related Products, Part 4: Optical Test, Section 1: Opacity [CHD 20: Paints, Varnishes and Related Products]

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Indian Standard (Reaffirmed 2004)

METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

PART 4 OPTICAL TEST

Section 1 Opacity

(Third Revision)

Paints and Allied Products Sectional Committee, CDC 8: Panel for Methods of Test, CDC 8: 6 : 4 [Ref : Doc : CDC 8 (9324)]

1. Scope — Prescribes three methods for determination of contrast ratio (opacity) of paints, varnishes and related products.

2. Method Using Black and White Charts

2.1 *Principle* — This method is based on the observation that contrast ratio is an approximately linear function of the reciprocal film thickness. Over a restricted film thickness range which also corresponds to that used for normal application of white or light coloured paints. It is thus possible to interpolate graphically or by computation between results on films of different thicknesses, with satisfactory accuracy.

2.1.1 Since wet film thickness cannot be determined with sufficient accuracy, the method involves determination of dry film mass per unit area and a calculation of the corresponding wet film thickness. In this latter calculation, values for wet film density and percentage of non-volatile matter content are required. Determination of these values by the methods complying with the relevant Indian Standards has been prescribed. It is, however, recognized that for certain types of paints, non-volatile determination as given in relevant Indian Standard may not exactly correspond to the mass changes of a film during drying under conditions of this test. Any errors in results introduced by this discrepancy should be common to all test laboratories and should not affect comparison of paints of similar types.

This standard prescribes a method for given paints applied at a spreading rate of 20 m^2/l to an agreed black and white substrate.

2.2 *Apparatus*

2.2.1 *Substrate* — Charts, all of same size and measuring at least 100 × 200 mm, printed and varnished to give adjacent black and white areas readily wetted by, but impervious to solvent or water-thinned paints.

The black and white areas shall each be of dimensions not less than 80 × 80 mm. The reflectance of white areas of the cards shall be 80 ± 2 percent when measured over a white tile using a reflectometer complying with **2.2.3** and that of the black areas shall be not greater than 1 percent, unless otherwise agreed.

To avoid errors due to variation, from one batch of charts to another, the charts used for a given test should come from the same batch.

2.2.2 *Film applicators* — A series of film applicators giving a range of uniform films of wet thicknesses approximately 40 to 60 μm is required. The film laid down shall be at least 70 mm wide, with areas of dimensions not less than 60 × 60 mm and of uniform thickness over both black and white areas of the card. The application of uniform films is facilitated by the use of automatic applicators.

2.2.3 *Reflectometer* — A photoelectric instrument giving within 0·3 percent of the indicated reading proportional to the intensity of light reflected from the surface under test, and having a spectral response approximating to the product of relative spectral energy distribution of CIE illuminant C or D 65 and the colour matching function $y(\lambda)$ of the CIE standard observer. The value measured is R_Y.

Note — It is recognized that the relative geometrical arrangement of the illuminating beam and the light detector can affect the measurement of R_Y but it is considered that variations arising from this factor in commercial reflectometers should be considerably less than the reproducibility figure expected. In the evento f dispute, 0°/diffuse geometry, excluding specular reflection, should be used.

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2.2.4 *Template or die stamp* — A metal template or die stamp of dimensions not less than 60 × 60 mm is suitable for accurate removal of a closely defined area from a test chart.

2.3 *Procedure*

2.3.1 *Preparation of substrate* — Store the black and white substrate charts, in single thickness, under the conditions of testing (27 ± 2°C and a relative humidity of 65 ± 5 percent) for at least 24 h before coating, handle them at all times by the edges to avoid finger marks on the areas to be coated. Weigh, to the nearest 1 mg, six charts for coating, and two charts to be kept as blank controls. Prepare the charts for coating by one of the following methods:

a) Fixing one end by clips or adhesive tape, to a flat glass plate at least 6 mm thick, or

b) Using a vacuum suction plate which should be flat to within ±2 μm, or

c) Fixing one end and laying it over a flat rubber block (where spiral applicators are to be used).

2.3.2 *Preparation of coated charts* — Immediately before application, mix the paint thoroughly by vigorous stirring to break down any thixotropic structure, taking care not to incorporate air bubbles.

Apply about 2 to 4 ml of paint according to the film thickness required, in a line across one end of the chart and spread it immediately by drawing down a suitable applicator at a steady velocity to give a uniform layer. Prepare duplicate films with each of three different applicators, chosen to give a range of wet film thicknesses from 40 to 60 μm approximately.

Maintain the coated charts in a horizontal position until dry, for example, by taping the edges to a flat substrate. The drying time (and/or stoving conditions) will depend on the type of paint material being tested, and should be agreed to by the interested parties.

2.4 *Conditioning* — Keep the dried coated charts and the blank charts at 27 ± 2°C and a relative humidity of 65 ± 5 percent for at least 24 h and not more than 168 h before the reflectance measurements are made.

2.5 *Measurement of Reflectance Factors* — Measure the reflectances of each coated chart at a minimum of four positions over both the black and white areas of each chart, and calculate the average reflectance factors R_B and R_W, respectively. Then calculate the contrast ratio R_B/R_W for each coated chart.

2.6 *Determination of Surface Density of the Dry Coating* — By means of metal template and a sharp knife or precision die stamp, cut equal areas of dimensions at least 60 × 60 mm, from the centres of the blank and the coafed charts. Weigh the detached pieces to the nearest 1 mg.

Calculate the surface density of the dry coating, Q_A in grams per square millimetre, by the formula:

$$Q_A = \frac{m_4 - m_3 \times \frac{m_2}{m_1}}{A}$$

where

m_1 = average initial mass, in grams, of the blank control charts;

m_2 = initial mass, in grams, of the chart to be coated;

m_3 = average mass, in grams, of the cut portions of the blank control charts;

m_4 = mass, in grams, of the cut portion of the coated chart; and

A = area, in square millimetres, of the cut portion of the chart.

Note — This technique eliminates the effect of changes in the masses of the charts due to variations in moisture content if it can be assumed that blank and coated charts change equally.

2.7 *Calculation of Wet Film Thickness and Spreading Capacity* — To calculate the wet film thickness from the surface density of the dry coating, it is necessary to know both the density of the wet paints, and the non-volatile matter content by mass.

2.7.1 *Wet film thickness* — Calculate the thickness of the wet paint film, t, in millimetres, using the formula:

$$t = \frac{Q_A}{Q \times NV} \times 10^5$$

where

Q = density of the paint, in grams, per millimetre; and

NV = non-volatile matter content, as a percent by mass.

2.7.2 *Spreading capacity* — The spreading capacity, *SC*, in square metres per litre is the reciprocal of wet film thickness, in millimetres, and is given by the formula:

$$SC = \frac{1}{t} = \frac{Q \times NV}{Q_A} \times 10^{-5}$$

and using the formula for surface density in **2.6**:

$$SC = \frac{A \times Q \times NV}{m_4 - m_3 \times m_2/m_1} \times 10^{-5}$$

2.8 *Determination of Contrast Ratio for a Spreading Capacity of 20 m^2/l* — It is assumed that for a limited range of film thickness, the contrast ratio is a linear function of the spreading capacity. Therefore, the values of contrast ratio and the corresponding spreading capacities obtained for each of the six films should be plotted graphically and the contrast ratio at a spreading capacity of 20 m^2/l determined by linear interpolation. The calculation can be made less laboriously, where facilities are available, by computing a regression of contrast ratio on spreading capacities from the experimental data.

3. Method Using Polyester Film

3.1 *Principle* — This method is also very similar to the one for determination of opacity by the method using black and white charts. This method prescribes the determination of opacity of given paint films applied at a spreading capacity of 20 m^2/l to colourless transparent polyester film, the reflectance being measured subsequently over agreed black and white glass plates.

3.2 *Apparatus*

3.2.1 *Substrate* — Untreated, colourless, transparent polyester film between 30 and 50 μm in thickness having refractive index value of between 1·64 to 1·67 and of dimensions not less than 100 × 150 mm. The use of thicker film is permitted by the agreement between the parties.

3.2.2 *Film applicators* — A series of film applicators giving a range of uniform films of wet thicknesses approximately 40 to 60 μm is required. The film laid down shall be at least 70 mm wide, with areas of dimensions not less than 60 × 60 mm and of uniform thickness over the polyester film. The application of uniform films is facilitated by the use of automatic applicators which are recommended.

3.2.3 *Reflectometer* — A photoelectric instrument giving within 0·3 percent of the indicated, reading proportional to the intensity of light reflected from the surface under test, and having a spectral response approximating to the product of the relative special energy distribution of CIE illuminant C or D65 and the colour matching function $y(\lambda)$ of the CIE standard observer. The value measured is R_y.

Note — It is recognized that the relative geometrical arrangement of the illuminating beam and the light detector can affect the measurement of R_y but it is considered that variations arising from this factor in commercial reflectometers should be considerably less than the reproducibility figure expected. In the event of dispute, 0°/diffuse geometry, excluding specular reflection, should be used.

3.2.4 *Template or die stamp* — A metal template or die stamp of dimensions not less than 60 × 60 mm is suitable for accurate removal of a closely defined area from a test chart.

3.2.5 *Test plates* — Black and white glass plates, each with a plane, polished surface, of at least 80 × 80 mm. The reflectance of the white plate shall be 80 ± 2 percent when measured using a reflectometer complying with **3.2.3** and that of the black plate not more than 1 percent.

Both the black and the white plates should be coated on the back and edges with light-excluding paint or adhesive tape.

3.3 *Procedure*

3.3.1 *Preparation of substrate* — Prepare the polyester film for coating by one of the following methods:

a) Spreading it on a flat glass plate, at least 6 mm thick, which has first been moistened with a few drops of white spirit just sufficient to hold the film in position by surface tension, ensure that none of the liquid wets the upper surface of the film and that no air-bubbles are trapped under it; or

b) Fixing it at one end and laying it over a flat rubber block (where spiral applicators are to be used).

3.3.2 *Preparation of coated films* — Immediately before application, mix the paint thoroughly by vigorous stirring to break down any thixotropic structure, taking care not to incorporate air bubbles.

Apply about 2 to 4 ml of paint, according to the film thickness required, in a line across one end of the polyester film, and spread it immediately by drawing down a suitable applicator at a steady velocity to give a uniform layer. Prepare duplicate films with each of three different applicators, chosen to give a range of wet film thicknesses from 40 to 60 μm approximately.

Maintain the coated films in a horizontal position until dry, for example, by taping the edges to a flat substrate. The drying time (and/or stoving conditions) will depend on the type of paint material being tested, and should be agreed to by the interested parties.

3.3.3 *Conditioning* — Keep the dried coated films at 27 $\pm$ 2°C and a relative humidity of 65 $\pm$ 5 percent for at least 24 h and not more than 168 h before the reflectance measurements are made.

3.3.4 *Measurement of reflectance factors* — Fix the coated film in turn over the white and the black glass plates, introducing a few drops of white spirit between the underside of the film and the glass to ensure optical contact. Measure the reflectances of each coated film at a minimum of four positions over both the black and the white plates, and calculate the average reflectance factors R_B and R_W, respectively. Then calculate the contrast ratio R_B/R_W for each coated film.

3.3.5 *Determination of surface density of the dry coating* — Remove the coated film from the glass plate, wipe the film from white spirit and allow it to dry. By means of the metal template and a sharp knife or precision die, stamp, cut equal areas of dimensions at least 60 $\times$ 60 mm from the centres of the coated polyester films. Weigh the detached pieces to the nearest 1 mg. Remove the paint film by the use of a solvent which has been found to have no effect on the dried mass of the polyester film, and after thorough drying, reweigh the film.

Note — Acetone or dichloromethane are usually suitable as solvents. In cases where the dry coating is resistant to them and a solvent with greater power is required, the effect of the solvent on the mass of uncoated polyster should be determined.

Calculate the surface density of the drying coating, Q_A, in grams per square millimetre, by the formula:

$$Q_A = \frac{m_2 - m_1}{A}$$

where

m_1 = mass, in grams, of the uncoated polyester film;

m_2 = mass, in grams, of the coated polyester film; and

A = area, in square millimetres, of the cut portion of the polyester film.

3.3.6 *Calculation of wet film thickness and spreading capacity* — To calculate wet film thickness from the surface density of dry coating, it is necessary to know both the density of the wet paint, and the non-volatile matter content by mass.

3.3.6.1 *Wet film thickness* — Calculate the thickness of the wet paint film, t, in millimetres, using the formula:

$$t = \frac{Q_A}{Q \times NV} \times 10^5$$

where

Q = density of the paint, in grams per millilitre; and

NV = non-volatile matter content, as a percent by mass.

3.3.6.2 *Spreading capacity* — The spreading capacity SC, in square metres per litre, is the reciprocal of the wet film thickness, in millimetres, and is given by the formula:

$$SC = \frac{1}{t} = \frac{Q \times NV}{Q_A} \times 10^{-5}$$

and using the formula for surface density in **3.3.5** above:

$$SC = \frac{A \times Q \times NV}{m_2 - m_1} \times 10^{-5}$$

3.3.7 *Determination of contrast ratio for a spreading capacity of 20m²/l* — It is assumed that. for a limited range of film thickness, the contrast ratio is a linear function of the spreading capacity. Therefore, the values of contrast ratio and the corresponding spreading capacity obtained for each of the six films should be plotted graphically and the contrast ratio at a spreading capacities of 20 m^2/l determined by linear interpolation. The calculation can of course be made less laboriously where facilities are available, by computing a regression of contrast ratio on spreading capacities from the experimental date.

4. Pfund Cryptometer Method

4.1 *Principle* — The cryptometer is an instrument designed to measure the wet opacity of the paint. It consists of a calibrated wedge and enables the operator to determine the thickness of the paint required for complete obliteration of a contrasting black and white surface. This can be converted to wet opacity per litre.

4.1.1 The shades for which white/black base is to be used are given in Appendix A.

4.2 *Apparatus*

4.2.1 *Pfund cryptometer* — The cryptometer as shown in Fig. 1 consists of the following parts:

a) *Base plate (A in Fig. 1)* — It consists of two glass plates, black (*B*) and white (*W*) fused together along the line *LM*.

The reflection coefficients for white and black parts of the base plate are 80 ± 2 percent and less than 8 percent, respectively. Along the two longer edges and perpendicular to the line *LM* there are two parallel shallow grooves. In the black as well as white glass plates, there are two scales in millimetres along the grooves with '0' graduation at the dividing line *LM* between the black and white halves. The upper surface of the base plate of both the black and the white glass is optically flat.

b) *Top plate (P in Fig. 1)* — It is made of clear glass with its lower surface optically flat.

When placed on the base plate, the lower side of the top plate facing the base plate has two small steel legs which form a wedge-shaped film of the paint between the base and the top plates. Two top plates with different wedge angle constants are supplied for use with (1) black and dark-coloured paints, and (2) white and light coloured paints.

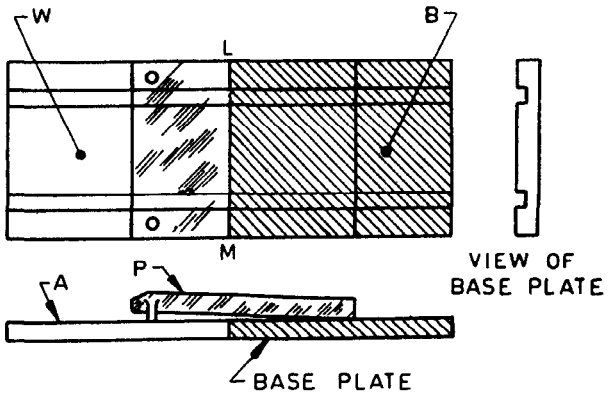

FIG. 1 PFUND CRYPTOMETER

4.3 *Procedure* — Place a few drops of the paint sufficient to fill the wedge-shaped clearance between the base plate and the top plate along the dividing line *LM* between the black and white halves of the base plate. Carefully place the top plate on the paint, eliminating bubbles. Gradually draw the top plate along the length of the base plate so that the dividing line just disappears. Note the scale reading at the point of contact with the top plate. Then move the top plate in the opposite direction till the line of demarcation reappears. Again record the scale reading at the point of contact with the top plate. Repeat the experiment ten times and calculate the mean for ten pairs of observations (that is, 20 readings) for the disappearance and reappearance of the dividing line *LM* of the black and white halves.

4.4 *Calculation*

$$\text{Wet opacity per litre} = \frac{1}{KL}\ \text{m}^2$$

where

K = wedge angle constant engraved on the top plate, and

L = mean scale reading.

APPENDIX A

(*Clause* 4.1.1)

PFUND CRYPTOMETER
CLASSIFICATION OF ISC SHADES FOR OPACITY DETERMINATION
PFUND CRYPTOMETER USING BLACK AND WHITE BASE PLATES

A-1. White Base

Sl No.	*Shade*	*ISC No.*	*Sl No.*	*Shade*	*ISC No.*
1.	Peacock blue	103	38.	Orange brown	439
2.	Azure blue	104	39.	Terra cotta	444
3.	Oxford blue	105	40.	Venetian red	445
4.	Aircraft blue	108	41.	Redoxide	446
5.	Navy blue	106	42.	Deep Indian red	448
6.	French blue	166	43.	Light purple brown	449
7.	Traffic blue	169	44.	Chocolate	451
8.	Grass green	218	45.	Gulf red	473
9.	Sage green	219	46.	Leaf brown	489
10.	Olive green	220	47.	Beech brown	490
11.	Brilliant green	221	48.	Service brown	499
12.	Light bronze green	222	49.	Fire red	536
13.	Middle bronze green	223	50.	Signal red	537
14.	Deep bronze green	224	51.	Post office red	538
15.	Light brunswick green	225	52.	Crimson	540
16.	Mid brunswick green	226	53.	Maroon	541
17.	Deep brunswick green	227	54.	Light orange	557
18.	Traffic green	267	55.	Traffic red	570
19.	Lincoln green	276	56.	India saffron	574
20.	Cypress green	277	57.	Deep orange	591
21.	Light olive green	278	58.	International orange	592
22.	Steel furniture green	279	59.	Silver grey	628
23.	Forest green	282	60.	Quaker grey	629
24.	Aircraft grey green	283	61.	French grey	630
25.	India green	284	62.	Light grey	631
26.	Scamic	294	63.	Dark admiralty grey	632
27.	Olive drab	298	64.	AF blue grey	633
28.	Middle buff	359	65.	Slate	634
29.	Deep buff	360	66.	Lead	635
30.	Middle stone	362	67.	Middle graphite	671
31.	Dark stone	363	68.	Smoke grey	692
32.	Light brown	410	69.	Aircraft grey	693
33.	Middle brown	411	70.	Dove grey	694
34.	Dark brown	412	71.	Dark blue grey	695
35.	Nut brown	413	72.	Black	—
36.	Golden brown	414	73.	Dark violet	796
37.	India brown	415			

A-2. Black Base

Sl No.	*Shade*	*ISC No.*	*Sl No.*	*Shade*	*ISC No.*
1.	Sky blue	101	16.	Light stone	361
2.	Torquoise blue	102	17.	Portland stone	364
3.	Oriental blue	174	18.	Vellum	365
4.	Eau-de-nil	216	19.	Light straw	384
5.	Sea green	217	20.	Light biscuit	385
6.	Opaline green	275	21.	Champagne	386
7.	Verdigris green	280	22.	Sunshine	387
8.	Apple green	281	23.	Beige	388
9.	Canary yellow	309	24.	Jasmine yellow	397
10.	Pale cream	352	25.	Light salmon pink	442
11.	Deep cream	353	26.	Salmon pink	443
12.	Primrose	354	27.	Light admiralty grey	697
13.	Lemon	355	28.	White	—
14.	Golden yellow	356	29.	Traffic yellow	368
15.	Light buff	358			

EXPLANATORY NOTE

This is one of a series of standards dealing with sampling and testing of paints, varnishes and related products. Because different operators using the same draw down device may obtain films differing significantly in thickness and that reproducible results can be obtained by determination of the contrast ratio corresponding to a precisely fixed spreading capacity by interpolation between measurements at two or more measured film thicknesses, a spreading capacity of 20 m^2/l (wet film thickness of 50μm) which is considered as an average for brush application of a free flowing paint on a smooth non-porous surface has been fixed. This standard supersedes clause **10** of IS : 101-1964 'Methods of test for ready mixed paints and enamels (*second revision*)'. National Test House, Calcutta shall be collecting the opacity values in due course and it shall be specified at the time of next revision.

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